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Technical Report No. 10

A HIGH-PRESSURE, ROLLING-BALL TYPE VISCOMETER

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May 1, 1965

INTRODUCTION

The purpose of this report is to describe the details of the design, calibration and performance of the high-pressure rolling-ball type viscometer used in our studies of transport processes in and the structure of aqueous electrolytic solutions.

The rolling-ball type viscometer lends itself so easily to high pressure work that, although other types of high pressure viscometers have been developed,[1] it has been used by a majority of investigators of the viscosity of fluids under high hydrostatic pressures.[2-14]

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1. See for example M. M. Kusakov, L. A. Konovalova, and A. A. Konstantinov, Chem. Absts., 61, 6417d (1964).
2. A. E. Flowers, Proc. Am. Soc. Test. Mat., 14, 565 (1914).
3. M. D. Hersey, J. Wash. Acad. Sci., 6, 525 (1916).
4. M. D. Hersey and H. Shore, Mech. Eng., 50, 221 (1928).
5. B. H. Sage, Ind. Eng. Chem., Anal. Ed., 5, 261 (1933).
6. F. Hoeppler, Z. Tech. Physik, 14, 165 (1933).
7. B. H. Sage and W. N. Lacey, Am. Inst. Mining Met. Engrs., Tech. Pub. 845 (1937).
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9. R. B. Block, J. Appl. Phys., 11, 635 (1940).
10. B. H. Sage and W. N. Lacey, Ind. Eng. Chem., 32, 587 (1940).
11. R. M. Hubbard and G. G. Brown, Ind. Eng. Chem., Anal. Ed., 15, 212 (1943).
12. E. M. Griest, W. Webb, and R. W. Schiessler, J. Chem. Phys., 29, 711 (1958).
13. S. E. Babb, Jr., and G. J. Scott, J. Chem. Phys., 40, 3666 (1964).
14. G. E. McDuffie, Jr., and M. V. Kelly, J. Chem. Phys., 41, 2666 (1964).

DESCRIPTION OF THE EQUIPMENT

a.

The Pressure-Producing Equipment

The high hydrostatic pressure producing equipment was designed by and purchased from Harwood Eng. Co., Inc., Walpole, Mass. This equipment is capable of producing controlled pressures up to 200,000 lbs/in^2. The pressure is measured with a Manganin cell and the hydraulic fluid is an organic ester, Univis P-38 (Humble Oil and Refining Co.). A detailed description of this part of the apparatus can be found in previous reports and publications.[15,16]

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15. R. A. Horne and G. R. Frysinger, "The Effect of Pressure on the Electrical Conductivity of Sea Water", Arthur D. Little, Inc., Project TRIDENT Technical Report No. 1270862 (August 1962), Bur. Ships Contract NObsr-81564 S-7001-0307.

16. R. A. Horne, and G. R. Frysinger, J. Geophys. Res., 68, 1967 (1963).

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b.

Temperature Control

The thermostatic bath was also described previously.[15] The temperature control within the bath was to within 0.05°C. Unfortunately, the viscometer itself was too long to fit into the bath. As a consequence the viscometer was mounted external to the bath and the transformer oil bath fluid was circulated by a centrifugal pump through a jacket surrounding the viscometer. The flow of cooling fluid was relatively slow due to the restricted space between the viscometer pick-up coils and the walls of the jacket. This relatively slow circulation resulted in a temperature difference between the two pick-up coils. In the experiments at the lower temperatures, where the differences between room and fluid temperatures was greatest, this difference could be as great as 0.5°C. The temperature was determined with copper-constantan single junction thermocouple at each of the pick-up coils and the average temperature used. This procedure proved quite satisfactory and yielded viscosities at one atmosphere and at different temperatures in

good agreement in the literature values.[17] Further attempts to improve the temperature control were therefore considered unnecessary.

17. R. A. Horne, R. A. Courant, D. S. Johnson, and F. F. Margosian, "The Activation Energy of Viscous Flow of Pure Water and Sea Water in the Temperature Region of Maximum Density", Arthur D. Little, Inc., Tech. Rept. No. 4 (Oct. 31, 1964), Office of Naval Res. Contract No. Nonr-4424(00).

c.

The Viscometer

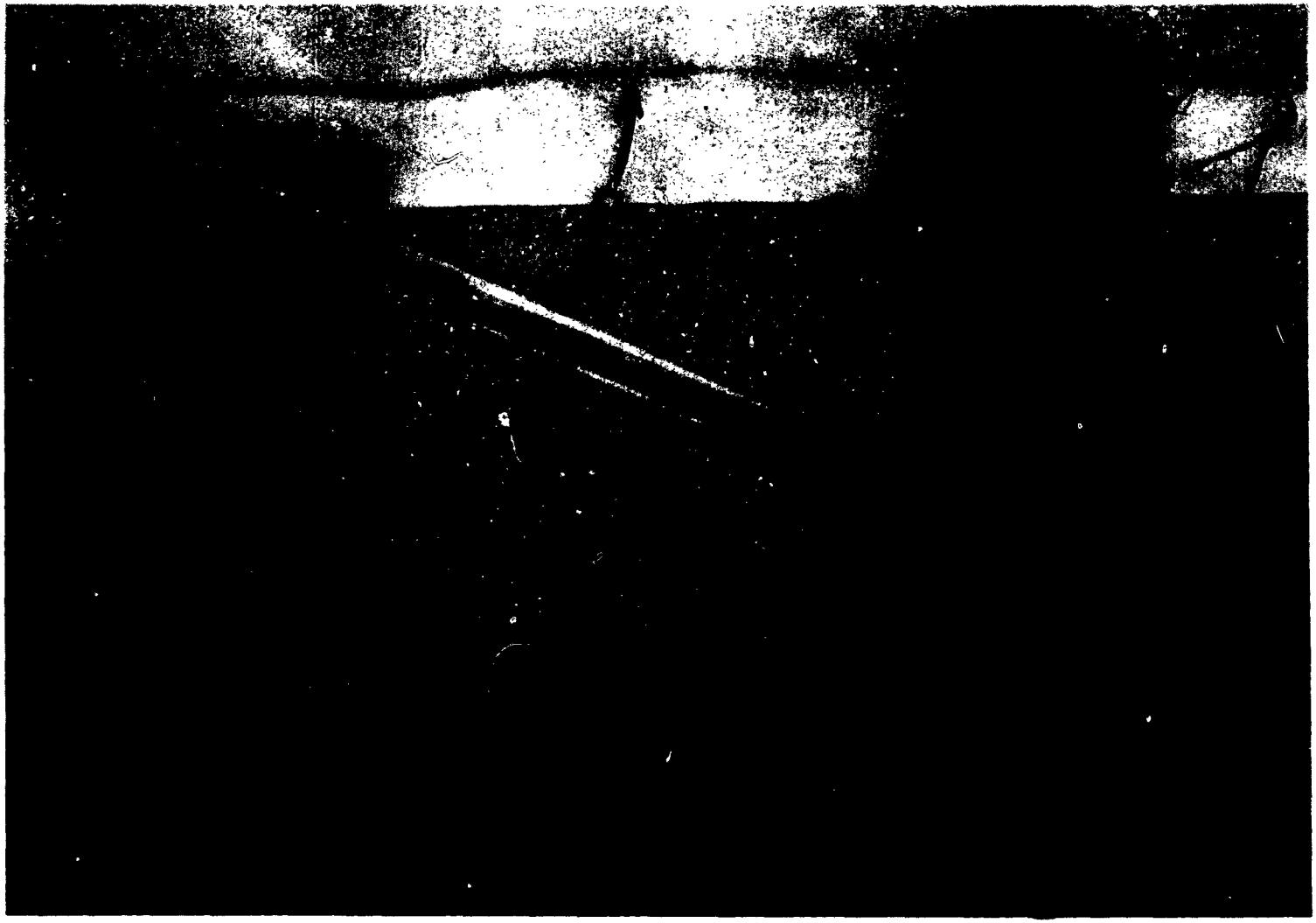
The high pressure rolling ball viscometer is shown in Fig. 1. The viscometer itself, V, consists of a length of 3/8" O.D., 1/8" I.D. stainless steel, nonmagnetic tubing 30" in length closed at one end with a dead end plug, P. The viscometer is enclosed in a lucite jacket, J, through which cooling fluid circulates from the inlet I to the outlet O. Two openings, T_1 and T_2, are bored through this jacket for the insertion of the thermocouple plugs (not shown). The leads, L, from the pick-up coils C_1 and C_2 are carried out through one end of the jacket. The whole apparatus is inclined at an angle θ and the steel rolling ball is raised to the top of the instrument with the magnet, M.

The viscometer design incorporates one important innovation. The ball rolls, not along the inside of the high pressure tubing itself but rather along a loose-fitting inner-lining consisting of thin wall stainless steel tubing, 0.0955 in. I.D., slightly crimped near the lower end to stop the ball's descent. Pressure forces the walls of the high pressure tubing outwards, thereby increasing the inside diameter, but there is no pressure differential across the walls of the inner liner, thus the ball moves in a tube of essentially constant diameter and there is no significant correction factor with increasing pressure.

In order to avoid corrosion initially the 0.0625 in. diameter balls were Ni-striked and Au-plated. After a short time the fall time of these balls became erratic and tended to increase. Microscopic examina-

FIGURE 1

High Pressure Rolling Ball Viscometer



tion revealed that the Au-plate had blistered. Ni-plated balls were substituted. These performed satisfactorily and were quite corrosion resistant. The balls were very uniform; no differences among the balls was detectable from viscometer fall times, weightings, or diameters as measured with a micrometer.

The angle, θ, used was 30° and this optimum value was determined by trial and error. A more steep inclination sacrificed a significant figure of the fall time, while less steep inclinations tended to give erratic fall times. The latter was probably due to the greater likelihood of a very slow moving ball getting retarded by dust motes or slight inhomogeneties on the inner surface of the liner.

d.

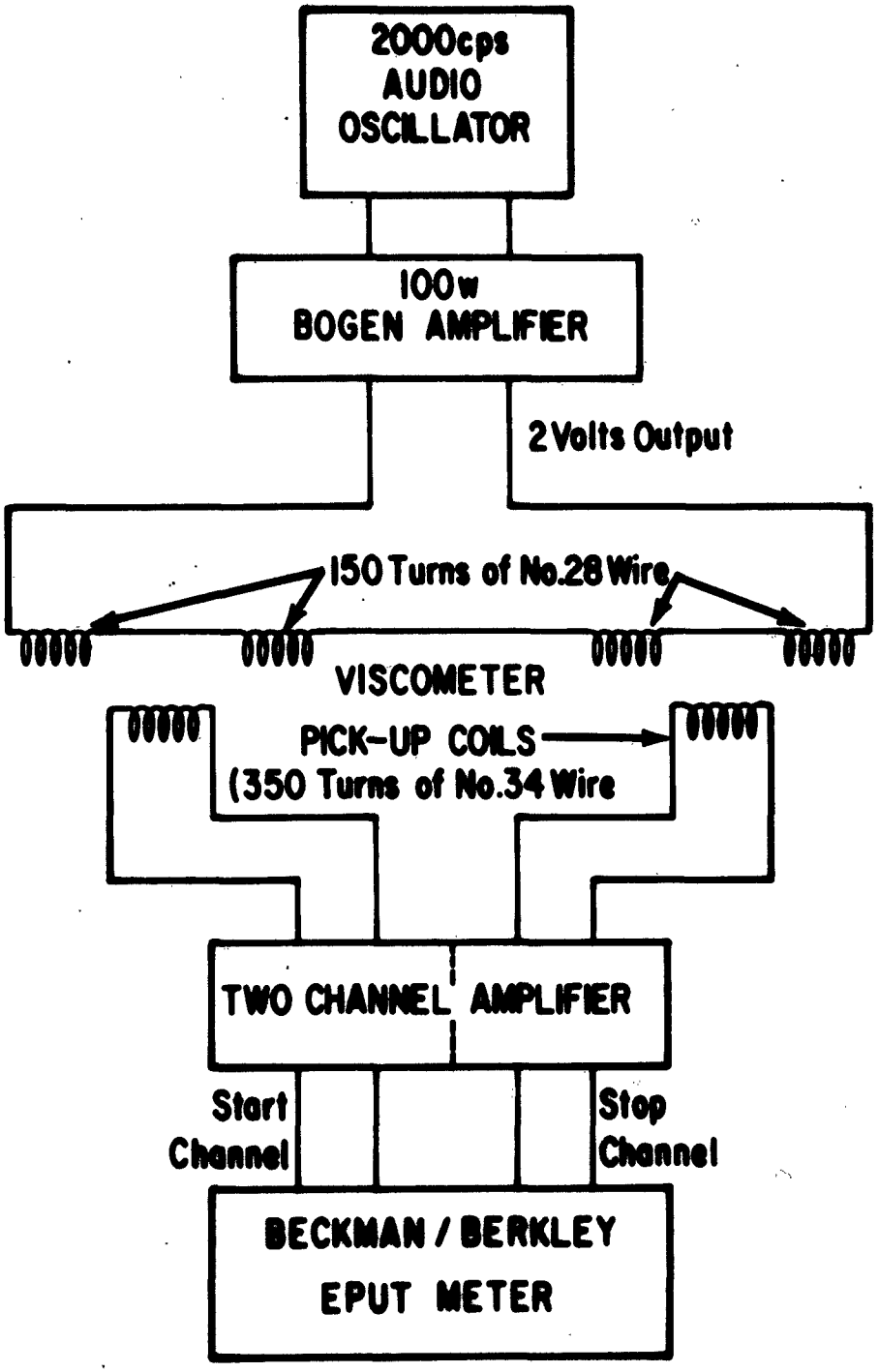
Timing Circuit

The rolling ball viscometer timing circuit is shown in Figure 2. Its operation is straightforward. Using a magnet, the steel ball is brought to the top of the inclined viscometer section. When the magnet is withdrawn, the ball rolls down the inclined section due to the gravitational force.

Two sets of coils are arranged a fixed distance apart (approximately 26 cm) along the viscometer. Each set of coils contains two primary sections, wound in series opposition, and a centrally located pick-up coil. The primaries are energized by an audio oscillator feeding 2,000 cycles to a 100 watt amplifier. The amplifier is adjusted so as to put out a two volt signal. (The D.C. resistance of each coil is approximately 6 ohms.) In the absence of the steel ball, the effect of the two primary sections on the pickup coil cancels out and the output voltage should be zero. Although each section of the primary coils has been designed to be exactly the same, a ten-millivolt background signal remains in each pair of coils due to residual unbalance in the mutual inductance. This is caused by slight differences in geometry when the coils are wound.

FIGURE 2

Viscometer Circuit Diagram


2000cps
AUDIO
OSCILLATOR
100w
BOGEN AMPLIFIER
2 Volts Output
150 Turns of No.28 Wire
VISCOMETER
PICK-UP COILS
(350 Turns of No.34 Wire
TWO CHANNEL AMPLIFIER
Start
Channel
Stop
Channel
BECKMAN / BERKLEY
EPUT METER

The EPUT meter contains a precision quartz crystal controlled clock. As the ball passes through the first set of coils, it produces a gross imbalance in the coupling and thus induces a voltage in the pickup coil. This signal is amplified and fed into the start channel of the EPUT meter. It triggers the start channel and actuates the running time readout. As the ball passes through the second set of coils, the process is repeated except that the amplified signal is fed into the stop channel of the EPUT meter and terminates the readout of the elapsed time. The interval of time between the start and stop signals is read directly off the digital display on the EPUT meter. Since the distance between the two pairs of coils is known and the time for the ball to roll between the two pairs of coils is measured, the velocity of the rolling ball can be calculated.

Operating Procedure

Immediately prior to a run the viscometer is disassembled and the high pressure tube (V), and its stainless steel liner rinsed successively with washes of 1) acetone, 2) distilled water, and 3) the solution to be studied. The liner is next inserted and a rubber tube attached to a filling funnel is fixed to the lower end of the viscometer. The viscometer is then filled by equalizing the levels in funnel and viscometer. The liquid in the viscometer is subjected to a mild vacuum to remove gases and the ball carefully inserted with the tube inclosed in such a manner to minimize gas entrainment. The viscometer is loosely connected to the high pressure system and a slight pressure applied until the escape of hydraulic fluid through the loose coupling indicates that the air space between the fluid to be studied and the hydraulic fluid is removed. The proper angle of the viscometer is checked with a special level, the connection tightened, and the level checked again. The angle is also checked in the course of the experiment since it is very critical.

Following each pressure increment the apparatus is allowed to stand from 10-15 minutes in order that the temperature increase resulting from adiabatic compression be dissipated. The ball is raised to the top

of the viscometer by hand with a 2495 gauss magnet with a 3.5 cm. gap. The ball is held at the top about 4-5 cm above the first pickup coil for about 30 sec. (longer for more viscous solutions). If this precaution is not taken the roll times tend to be erratic and not reproducible. Apparently this pause allows eddies set up by raising the ball to die away. The ball is then released and its fall time between the two pickup coils measured. The procedure is then repeated. A dozen or more rolls are made at each temperature and are alternated with temperature checks.

The initial position of the ball at the top of its roll is crucial. If the ball is too close to the top coil it may still be accelerating while in the timed zone between the two coils. On the other hand, if it is too near the top of the viscometer it may come in contact with the interface between the fluids. At 10,000 atm. this interface has moved at least 16 cm down the tube due to the compressibility of the solution and, due to deformation of the viscometer walls, the actual distance moved is probably even greater. Fortunately, when the ball does accidently come in contact with the interface, erratic results obtain so the difficulty is easily recognized. A well designed viscometer should have an ample reservoir above the ball's initial position.

At a given temperature and pressure an experiment was repeated from 4 to as many as 10 times. In the course of a given series of runs the measured average deviation of the temperatures of the viscometer ranged from $\pm$ 0.005°C (for temperatures near room temperature) to as much as $\pm$ 0.02°C (for the lowest temperatures). Notice that these temperature deviations are less than that quoted above for the thermostatic bath because a given set of runs is completed in 10-15 min. whereas the temperature fluctuations in the bath range over periods from 1/2 to as long as several hours.

The ball roll time is read to four significant figures and the roll times range from 5-10 sec. The average deviation in the measured roll-time is about 0.10%.

Corrections

Over the temperature interval 0 to 10°C thermal expansion of the vitreous silica spacer between the pick-up coils increases the distance between them and thus the roll time by approximately 0.008%. The diameter of the rolling ball increases by about 0.034% but the effect of this increase is partially compensated by an approximately 0.018% increase in the diameter of the inner tube in which the ball rolls. These effects of thermal expansion are well within the measured average deviation of 0.10%; therefore no attempt is made to apply corrections for them. However, the temperature dependencies of the densities of the ball and of the fluid are taken into consideration (see below).

The application of pressure does not appreciably alter the spacing between the pick-up coils or the inside diameter, D_T, of the liner, and its effect on the diameter of the ball, D_B, is slight. The expressions derived by Hubbard and Brown[11] contain the terms D_B/D_T and $(D_T + D_B)$ and over the pressure range 15 to 150,000 lbs/in^2 they change by only 0.02 and 0.06% respectively. However, again, the pressure dependencies of the densities of the ball, ρ_B, and of the fluid, ρ_F, are taken into consideration.

Calibration and Data Analysis

In the present work the simplified relation developed by Sage[5] was used

1) $$\eta = C\, t\, (\rho_B - \rho_F)$$

where η is the viscosity, C is a constant, and t is the roll time. Over the temperature and pressure ranges involved the density of the steel ball is very nearly a constant but the density of water and thus of $(\rho_B - \rho_F)$ varies significantly. The density of steel, ρ_{Fe}, was calculated from the expression

2) $$\rho_B = \rho_{Fe} = 7.8835/(1 + 3.5 \times 10^{-5}T)\ (1-6.1 \times 10^{-7}\,[P-1]\,)$$

where T is the temperature in °C, P is the hydrostatic pressure in atmospheres and the constants are based on the densities, compressibilities, and coefficients of cubic thermal expansion from The International Critical Tables.

Figure 3 shows that up to about 40,000 lbs/in^2 there is good agreement between the densities of water as reported by Amagat[18] and Bridgman[19].

18. E. H. Amagat, Ann. Chim. et phys., 29, 68, 505 (1893).
19. P. W. Bridgman, Proc. Am. Acad. Arts Sci., 48, 307 (1913).

In view of this agreement, reliance was placed in Dorsey's compilation of specific volumes[20] and for a given P and T densities were calculated from specific volumes read or interpolated from Dorsey's Table 95. Values of $(\rho_B - \rho_F)$ at 0, 5, 10, 15, 20, and 25°C are given in Figure 4.

20. N. E. Dorsey, Properties of Ordinary Water - Substance, Reinhold Pub. Corp., New York, N. Y., 1940.

A calibration curve of the viscosity versus t $(\rho_B - \rho_F)$ (Figure 5) for aqueous NaCl solutions, using data of Sheely[21]; and water sucrose solutions using data of Bingham and Jackson[22] and more recent data of Swindells, Snyder, Hardy and Golden;[23] shows that the region of interest

21. M. L. Sheely, Ind. Eng. Chem., 24, 1060 (1932).
22. E. C. Bingham and R. F. Jackson, Bull. Nat. Bur. Stds., 14, 59 (1918).
23. J. F. Swindells, C. F. Snyder, R. C. Hardy, and P. E. Golden, Suppl. to Nat. Bur. Stds. Cir. No. 440 (July 31, 1958).

in the present experiments the curve is linear, thus equation (1) is applicable, and the following useful relationship is valid:

$$3) \quad \eta_p / \eta_{1 \text{ atm.}} = t_p \, (\rho_B - \rho_F)_p \; / \; t_{1 \text{ atm.}} \, (\rho_B - \rho_F)_{1 \text{ atm.}}$$

Initial Results

Dorsey[20] has summarized earlier literature on the viscosity of compressed water and it is possible to make a comparison between the values he quotes and some initial results obtained with the above described viscometer at 10°C. The three earlier investigators quoted,

FIGURE 3

Density of Pure Water Under Pressure

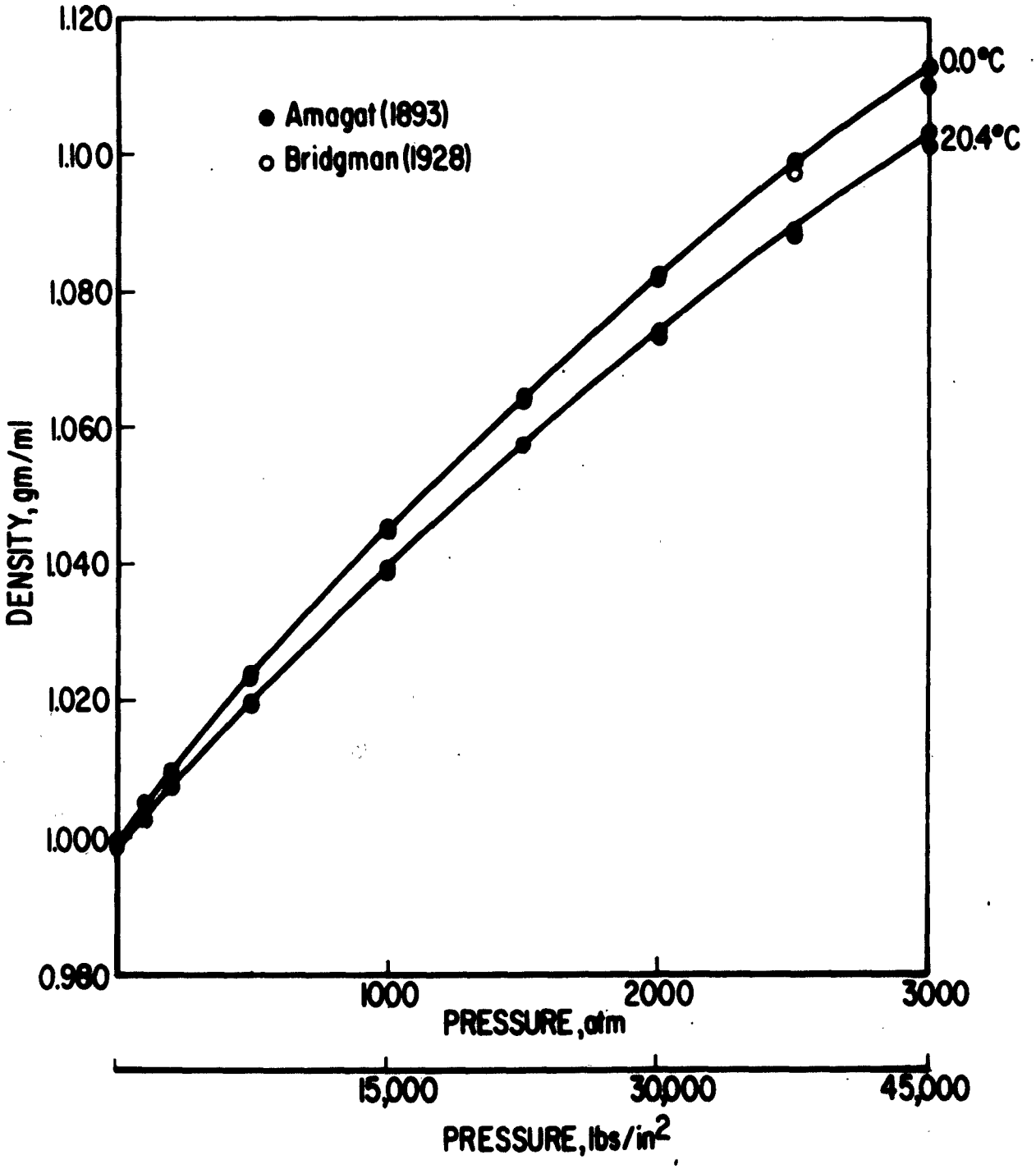

1.120
1.100
1.080
1.060
1.040
1.020
1.000
0.980
DENSITY, gm/ml
● Amagat (1893)
○ Bridgman (1928)
0.0°C
20.4°C
1000
2000
3000
PRESSURE, atm
15,000
30,000
45,000
PRESSURE, lbs/in²

FIGURE 4

Density Difference of the Rolling Ball and Pure Water

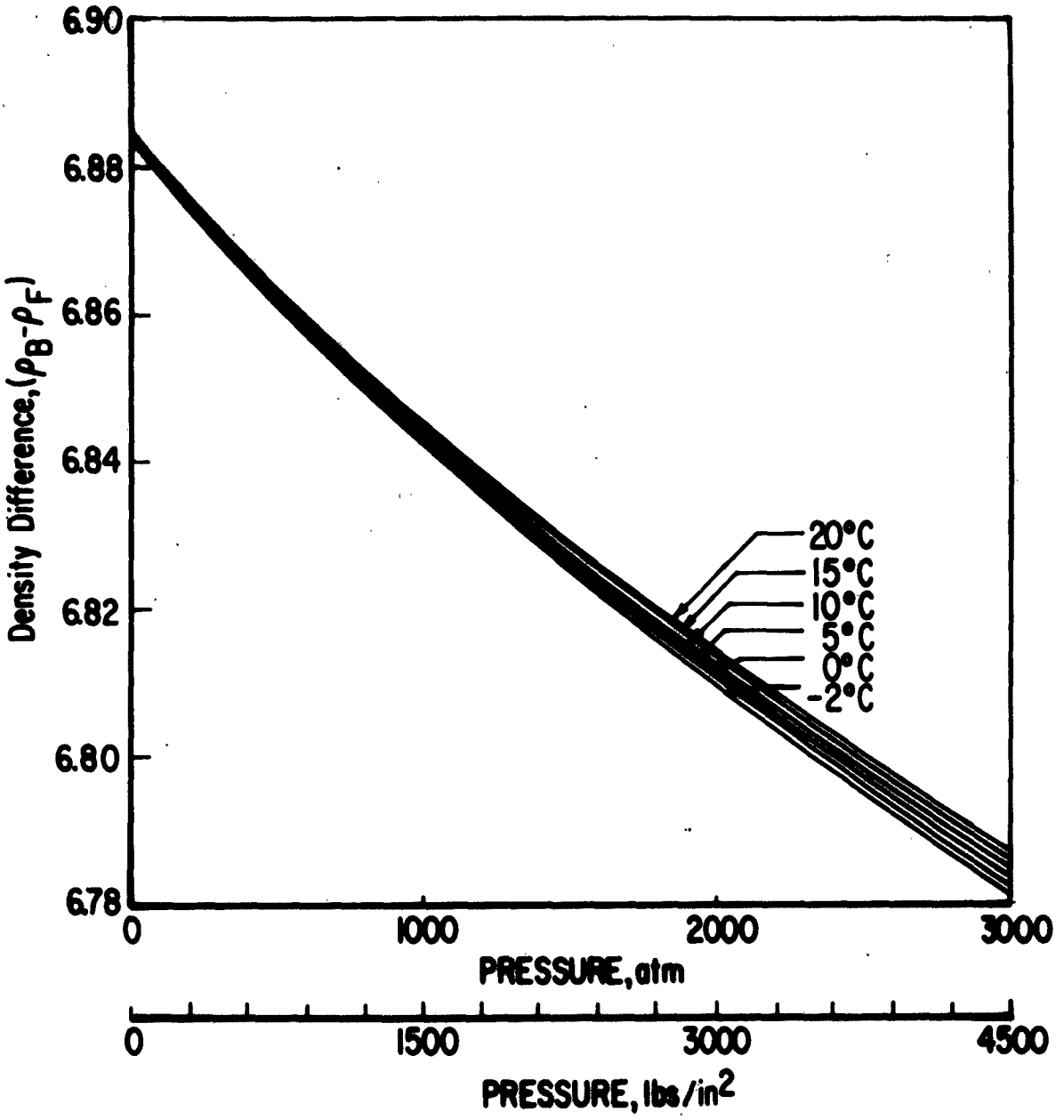
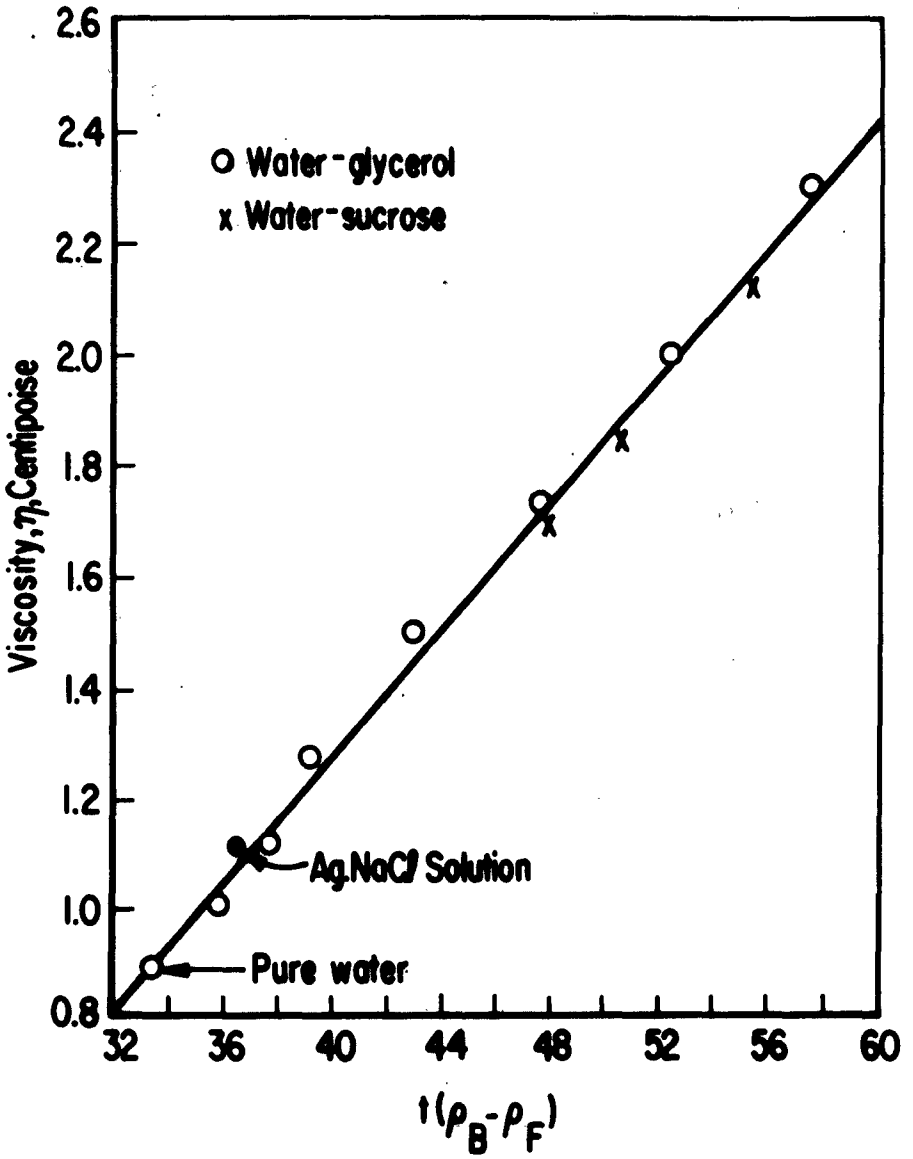

6.90
6.88
6.86
6.84
6.82
6.80
6.78
Density Difference, $(\rho_B - \rho_F)$
20°C
15°C
10°C
5°C
0°C
-2°C
0
1000
2000
3000
PRESSURE, atm
0
1500
3000
4500
PRESSURE, lbs/in^2

FIGURE 5

Calibration Curve


2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
Viscosity, η, Centipoise
O Water-glycerol
x Water-sucrose
Ag.NaCl Solution
Pure water
32
36
40
44
48
52
56
60
t(ρB-ρF)

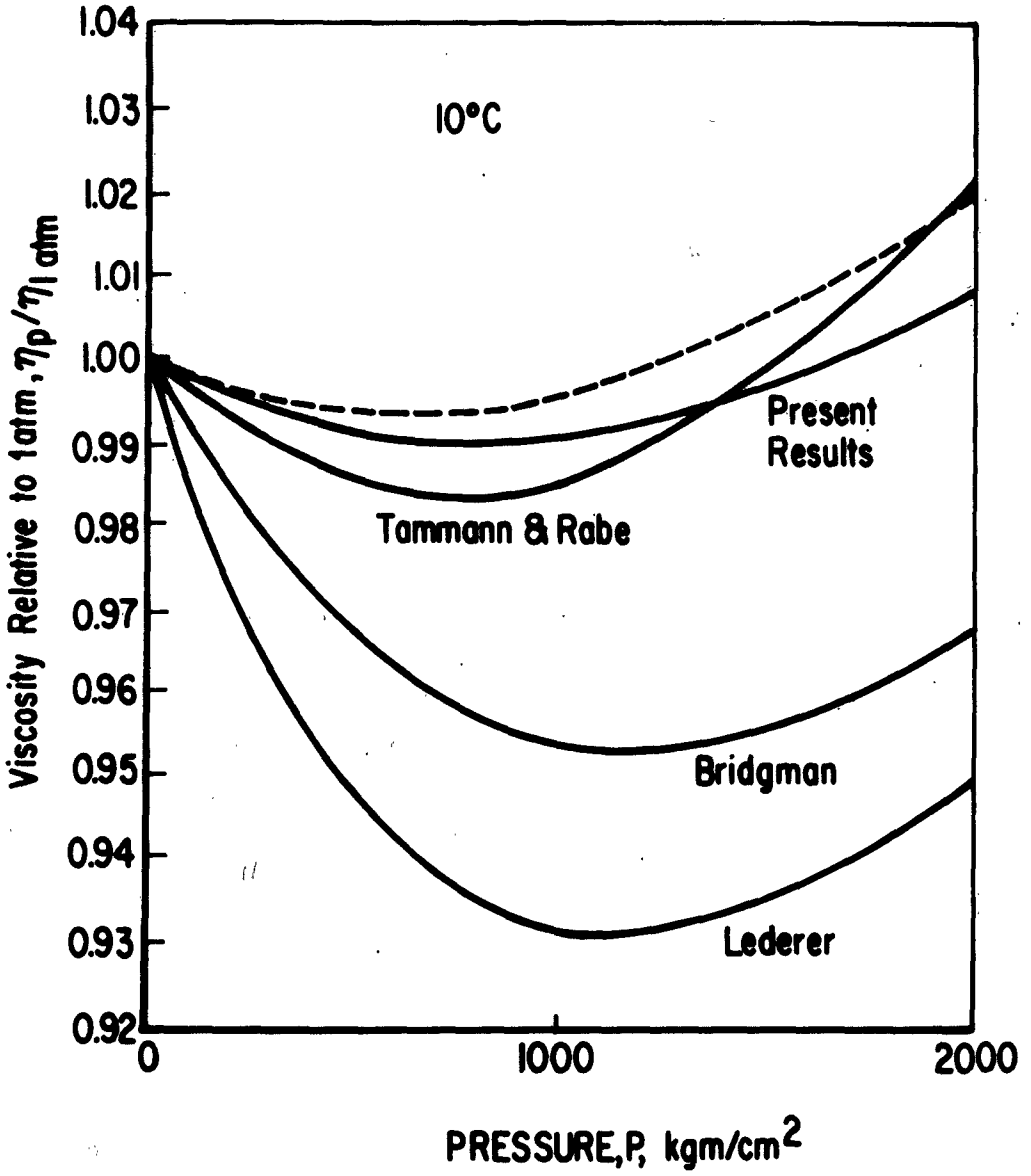
Bridgman[24], Tammann and Rabe[25], and Lederer[26], are not in agreement with one another (Figure 6). However, relative viscosities obtained with the

24. P. W. Bridgman, Proc. Am. Acad. Arts Sci., 61, 57 (1926); Prac, Nat. Acad. Sci., 11, 603 (1926).
25. G. Tammann and H. Rabe, Z.anorg. allgem. chem., 168, 73 (1927).
26. E. L. Lederer, Koll. Beih., 34, 270 (1932).

present apparatus do appear to be in fair agreement with Tammann and Rabe[25]. A plot of $t_p/t_{1\ atm.}$ (dashed line in Figure 6) shows that the density difference correction factor, $(\rho_B - \rho_F)_p / (\rho_B - \rho_F)_{1\ atm.}$ is much too small to account for the observed discrepancies between the present results and Bridgman[24] and Lederer.[26] The scale of the ordinate in Figure 6 is fairly expanded, thus at 1000 kgm/cm^2 the present results are about 0.5% higher than Tammann and Rabe[25] and about 5 and 7%. higher than Bridgman[24] and Lederer[26] respectively.

FIGURE 6

Comparison of Results


1.04
1.03
1.02
1.01
1.00
0.99
0.98
0.97
0.96
0.95
0.94
0.93
0.92
10°C
Present Results
Tammann & Rabe
Bridgman
Lederer
Viscosity Relative to 1atm, $\eta_p/\eta_{1\,atm}$
0
1000
2000
PRESSURE, P, kgm/cm^2

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The design, operational procedures, and performance of a rolling-ball type high pressure viscometer are described.

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Viscometer, pressure, viscosity, water.						

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14. **KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, roles, and weights is optional.

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